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AND

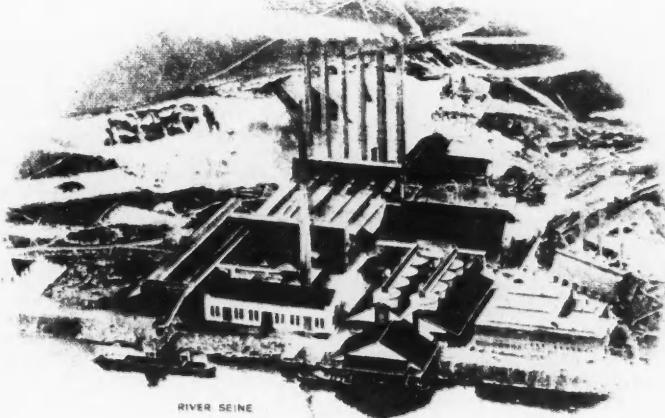
## CEMENT MANUFACTURE

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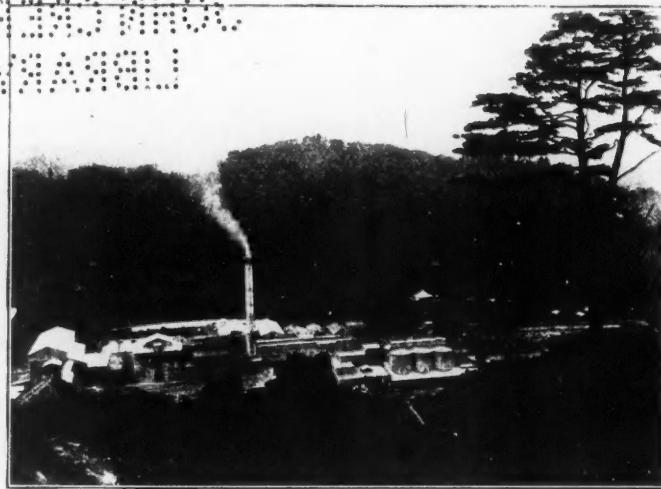
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# CEMENT AND CEMENT MANUFACTURE

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## Modification and Calibration of Kühl's Sedimentation Apparatus.

By COUNT CZERNIN (BERLIN).

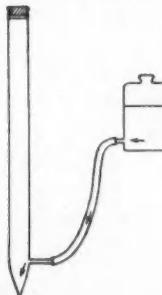
IN CEMENT AND CEMENT MANUFACTURE for November, 1932, Mr. K. Koyanagi describes work which he has carried out on the Kühl-Czernin sedimentation apparatus. He has gone to the trouble of calculating the theoretical sedimentation times in this apparatus of the individual fractions of different particle size by means of Stokes's law. This is a most difficult task, since the various sections of the sedimentation tube are at different temperatures, so that the values of viscosity and specific gravity are different. These theoretical calculations are extremely welcome, because the calculated values can now be compared with the empirical figures obtained practically, thus supplying a criterion for the exact method of using the apparatus. Actually, Koyanagi's work demonstrates that the results obtained practically scarcely differ from those obtained by calculation, and thus that the apparatus is well suited for exact measurements.

In the course of his work Koyanagi made certain modifications to the Kühl-Czernin apparatus to avoid certain defects which he found; the causes of which he attributed chiefly to the construction of the charging and discharging arrangements. It would appear, however, that these apparent defects were due to misunderstanding and that the apparatus was not used entirely correctly. That this is so seems to be confirmed by the greater simplicity and effectiveness of the original apparatus—at any rate as regards the discharging arrangement—when compared with Koyanagi's construction.

Koyanagi is in error when he states that the alcohol column in the Kühl-Czernin apparatus slowly sinks during the sedimentation and thus issues from the tube in drops which remove the cement particles from the apparatus. The volume of the alcohol column remains unchanged and is prevented from flowing out of the tube exactly as in Koyanagi's modification. The top opening of the

tube is closed by an air-tight rubber stopper, and the slow stream of alcohol which removes the sedimented cement grains from the apparatus is introduced from a reservoir which feeds in just above the exit from the tube, as is clearly shown in the figure. This arrangement appears to be superior to Koyanagi's modification, first, because it simplifies the manipulation of the apparatus—on passing from one fraction to the next a small turn of the filter stand is all that is necessary—and second, because it is advantageous to avoid rubber connections on the sedimentation tube, since the falling cement tends to adhere to surfaces which are not quite smooth.

The object of Koyanagi's modifications to the charging apparatus is to prevent settling of the cement in the container. For this purpose a continuous stream of air maintains the suspension in turbulent motion. The charging apparatus further contains a thermometer for controlling the temperature, since it is important that the temperature of the suspension should be the same as that of the alcohol which surrounds it. In the Kühl-Czernin apparatus this is attained by hanging the charging apparatus in the sedimentation tube during



the heating period, so that it is heated to exactly the same temperature as its surroundings. The continuous agitation of the suspension can be avoided by removing the container from the tube immediately before the sedimentation begins and thoroughly shaking it.

Koyanagi is correct in stating that one of the greatest disadvantages of the sedimentation method is the fact that it must be used with absolute alcohol. For this reason I have attempted to follow the Americans in using a defined mixture of petroleum fractions as the sedimentation liquid. Very good results can be obtained with a mixture of three parts petrol and one part medium oil, e.g., Russian watchmakers' lubricating oil. There is no conglomeration of the cement particles, and as a result of the higher viscosity the sedimentation proceeds more smoothly, although more slowly. The disadvantage of such a mixture is that it is not an individual chemical compound and that the apparatus must therefore be calibrated afresh whenever a different petroleum distillate is used. On the other hand the advantages of such a liquid are so great that it would be satisfactory if it were introduced into practice, particularly as Koyanagi has given so valuable an indication of the manner of carrying out such calibrations.

## Essential Flour in Cement.

THE following paper, by Mr. S. Rordam, is reproduced here by courtesy of Rock Products, in which journal it originally appeared on July 30th, 1932:

In an article in Rock Products for April 9, 1932, Mr. Eduardo Taylor assumes that the important flour in cement is the material ranging in grain size from  $10\mu$  to  $25\mu$ . This is based on the opinion of Wright<sup>1</sup>. Pearson and Sligh<sup>2</sup> consider the material finer than  $25\mu$  as flour. The Associated Portland Cement Manufacturers, Ltd., have adopted a standard elutriator to determine the "standard flour"<sup>3</sup> in cement. The material removed by an air current having a velocity of 21 ft. per minute is considered as flour. This flour has an average particle size of  $10\mu$ .

Helbig<sup>4</sup> points out that the strength, and especially the early strength, of cement increases with increasing fineness. He contends that there is no defined limit of fineness beyond which the strength begins to fall off. The imperviousness of the concrete made with the cement has also a direct relation to the amount of very fine flour in the cement.

Helbig's views are not shared by Kühl<sup>5</sup>, who is of the opinion that extreme fineness may cause a lowering of the strength. Kühl<sup>6</sup> has conducted an extensive study of air-separated fractions of cement. Using a von Gonell separator he split a cement up into seven fractions, each fraction differing from the next in grain diameter by  $10\mu$ . The individual fractions were tested for tensile and compressive strengths of 1:3 mortar by the miniature testing method developed by Kühl<sup>7</sup>. His test results led him to the conclusion that a cement having uniform grain size of about  $30\mu$  would have about the same strength as the original cement, and would require less power to grind on account of the smaller "inner surface." By reducing the grain size to 15-20 $\mu$  the strength would be increased considerably above that of the original cement, and it should still be within the limits of economical production to make such a cement. It would be entirely uneconomical to make cement finer than  $10\mu$ . The power consumption for the grinding would be beyond all reason, and the strength of such a fine cement would not be improved to any marked degree.

The tests upon which Kühl bases his conclusions, however, do not readily give a reliable picture of the concreting values of the various cement fractions. It has been shown by several investigators that the standard tests for tensile and compressive strengths of a 1:3 mortar are not really reliable criterions of how the cement is going to act when used in concrete, and when dealing with cement

<sup>1</sup> H. G. Wright, Further Development in Closed-Circuit Grinding. *Rock Products*, December 20, 1930.

<sup>2</sup> Pearson and Sligh, Bureau of Standards Technical Paper No. 48.

<sup>3</sup> A. C. Davis, Fineness and Sieving of Portland Cement, CEMENT AND CEMENT MANUFACTURE, February, 1932.

<sup>4</sup> A. B. Helbig, Feinzenement. *Zement*, No. 4, January, 1931.

<sup>5</sup> H. Kühl, Feinzenement. *Zement*, No. 8, February, 1931.

<sup>6</sup> H. Kühl, Der Einfluss des Feinkornaufbaues auf die Festigkeiteigenschaften der Portland-zement. *Zement*, No. 26-27, 1930.

<sup>7</sup> H. Kühl, Tonind. Ztg., 1929, p. 374.

having a granulometric composition quite different from that of ordinary cement this uncertainty of testing methods becomes very pronounced. Furthermore, this type of testing completely fails to give information concerning the plastic quality of the cement, a feature equally as important as strength when gauging the concreting value of a cement.

Experience has taught us that if a cement of a granulometric composition as obtained by present grinding methods satisfies certain standard tests it is reasonably certain that it will produce a concrete of satisfactory nature and durability. But if the granulometric composition of the cement is entirely changed, then the standard tests will fail to have any true significance. Although the specially graded cement may satisfy the present standard tests in a seemingly acceptable manner, the concrete made with the cement in question may not be satisfactory.

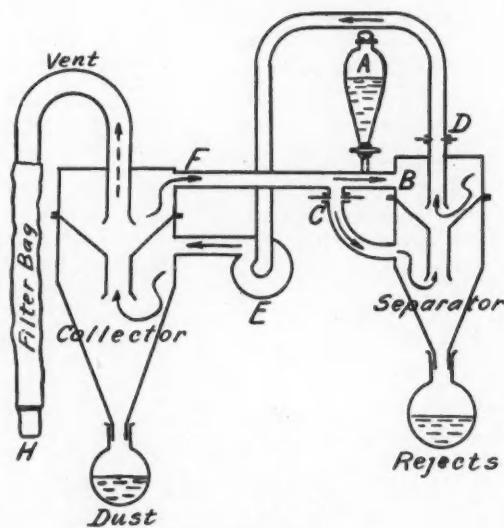


Fig. 1.

The test results obtained with cement graded  $10\text{-}25\mu$  (given later) furnish a good example of how cement having a good strength may lack the properties essential for making good concrete.

The definition of what may be considered the "essential flour" in cement has always been rather vague, and it must remain so unless it can be proved by tests that the important qualities of a cement can be referred to a particular interval of grading. Leaving out considerations of a chemical nature, the two most important qualities of cement may be said to be the power to confer plastic workability on the concrete mix and the bonding strength developed by the cement. If it can be proved that these two qualities are prevalent in a particular

interval of grading, one would have a ready answer to the question, "What is the essential flour in cement?"

The writer has done considerable work on fractionated air-separation of cement, and in his opinion it is the cement finer than  $10\mu$  which is really important. Early strength is largely determined by the content of flour finer than  $10\mu$ , and the important quality of plasticity and the power to "hold" the water in the freshly made concrete mix is almost entirely determined by the material finer than  $10\mu$ . This statement is confirmed by the experiments on air-separated cement carried out by Jennings\*. Using an air-separator similar to the one used by the writer, Jennings separated an ordinary Portland cement into various fractions and tested the chemical and physical qualities of the cements so obtained. The tests made by the writer were all completed before he read the paper by Jennings. It is gratifying to note how close an agreement there is in the analytical results obtained. The strength tests made by Jennings were unfortunately made on fractions separated from cement containing added gypsum, so the amounts of  $\text{SO}_3$  in his samples vary considerably. This introduces a factor of uncertainty in the tests for strength, as an increasing content of gypsum has a distinct influence on early strength.

In the tests carried out by the writer on air-separated samples of cement a Federal laboratory air separator was used (Fig. 1), the unit consisting of a separator and collector. The cement is introduced through the funnel A and is carried into the separator at B. In the separator centrifugal force throws the cement out against the wall where it drops through the inner cone. In the cylindrical extension of this cone the whirling cement meets an opposing air current blowing upwards and whirling against the cement. The "mesh diaphragm" at C controls the velocity of this opposing air current, which again determines the size of the material carried out of the separator. The coarse material drops into the flask for rejects. The fine dust carried out of the separator at D passes through the fan E and is blown into the collector. Here the dust is thrown out of the air current by centrifugal force, and falls into the flask at the bottom of the collector. The air current returns to the separator through F. A small amount of very fine dust escapes through the vent pipe. It is caught in the filter bag and collected in a beaker at the end of the bag H.

During the preliminary work with the separator it was found necessary to alter the original feeding device and replace it with a separatory funnel of glass so as to have an air-tight system of feeding the cement into the separator. The original feeder allowed too much air to leak into the system, and made it impossible to get a clean and sharp separation. Prior to the main test the separator was calibrated by determining the diaphragm combination which would give the desired fineness of separated material, and by a long series of tests a satisfactory working technique was established. It was found that the separation of the cement was easiest with the finest material up to a grain size of about  $25\mu$ .

\* J. H. Jennings, Properties of Portland Cement Particles Smaller Than 10 Microns. CEMENT AND CEMENT MANUFACTURE, March, 1932.

It was much more difficult to get a sharp separation of the material coarser than  $25\mu$ , as the relatively high air velocity in the separator tends to carry too much oversize. By combining the air-separation of the coarser material with a special sieving on an inclined 200-mesh sieve it was possible to get a very fair separation of the two fractions graded  $25-50\mu$  and coarser than  $50\mu$ .

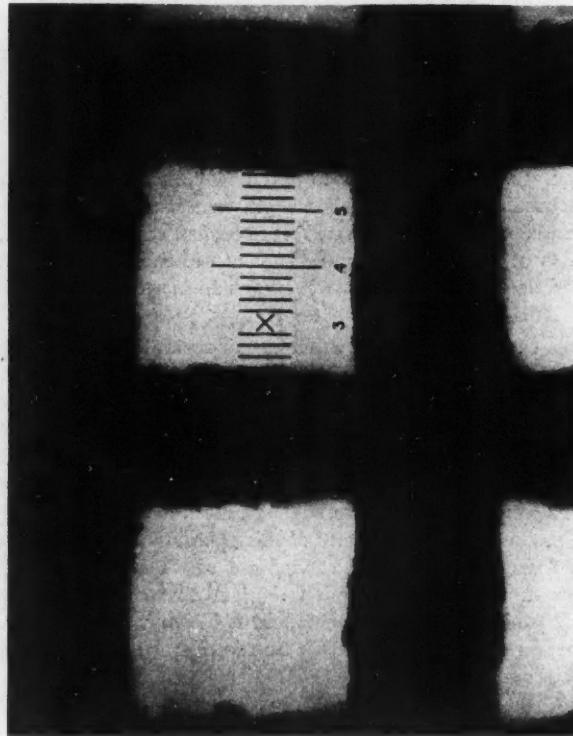


Fig. 2.—Photomicrograph of 200-mesh sieve magnified to the same degree as the photographs of the cement residues.

For all preliminary tests, which aimed at establishing the best working technique, a sample of regular Portland cement was used. This cement was ground in a 7 ft. by 26 ft. compeb mill to a fineness of 13 per cent. retained on the 200-mesh sieve. Repeated tests, with a very satisfactory agreement, gave the following granulometric composition of the cement: 17 per cent., plus  $50\mu$ ; 33 per cent.,  $25-50\mu$ ; 27 per cent.,  $10-25\mu$ ; 23 per cent.,  $0-10\mu$ . In calibrating for particle size one has to bear in mind that the grains are not of a uniform shape. The upper size limit of a particular fraction is therefore not determined by simply measuring what appears to be the largest particles. If all the cement

grains were cubes or spheres it would be a simple matter to determine the range of the grading. But a large number of the particles are flat or elongated, and may look like oversize material. For example, if a cube with sides of  $25\mu$  is compressed so that one dimension is reduced to one-half of its former size, the ensuing particle will be a flat prism measuring 12.5 by 35 by  $35\mu$ . In the microscope it will appear as a grain with a diameter of  $35\mu$ , but it is a perfectly legitimate particle in a grading having an upper size limit of  $25\mu$ , and it is a more reactive grain than a cubic particle as its surface is 13 per cent. greater than that of the cube. In measuring the grain size of a fraction it is therefore necessary to pay close attention to the shape of the particles until the eye learns to judge the particle size representing the true upper limit of the grading.

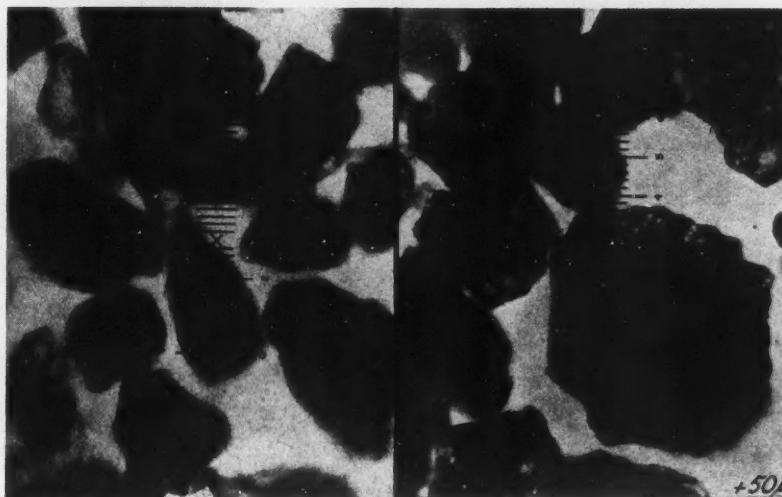


Fig. 3.—Fractions coarser than 50 microns. This closely represents the material retained on a 325-mesh sieve. Cement ground to a fineness of 80 to 90 per cent. passing a 200-mesh sieve contains 20 to 25 per cent. of this grading.

Fig. 2 is a microphotograph of a 200-mesh testing sieve, and Figs. 3 to 5 are photographs of the air-separated cement fractions from the main test. All are magnified to the same degree, about 425 times. On each photograph will be seen an imprint of the eyepiece micrometer in the microscope. With the lens combination used for the photographs each division of the eyepiece micrometer is equal to  $4.18\mu$ . A 200-mesh sieve has a nominal mesh opening of  $74\mu$ . By comparing the mesh opening with the grain diameters of the finer sizes, one gets a very striking idea of how little relation there is between a fineness test on a 200-mesh sieve and the fineness of the particles forming the really active part of the cement. This uncertainty of the fineness test on the 200-mesh sieve as a guide to the flour content is even more pronounced with air-separators in

closed-circuit with the grinding mills. The test for "standard flour" adopted by the Associated Portland Cement Manufacturers, Ltd., is a long step forward, but it seems to the writer that the interval of grading represented by "an average particle size of  $10\mu$ " is too large to give also a reliable measure of the amount of material finer than  $10\mu$ .

#### Grading and Distribution of Gypsum in Cement.

Owing to the easy grindability of gypsum and to its lower specific gravity as compared with cement clinker, it was to be expected that most of the gypsum would be found in the finest of the air-separated fractions. A sample of regular cement was split up into four fractions and each fraction tested for  $\text{SO}_3$  content with the results shown in Table I.

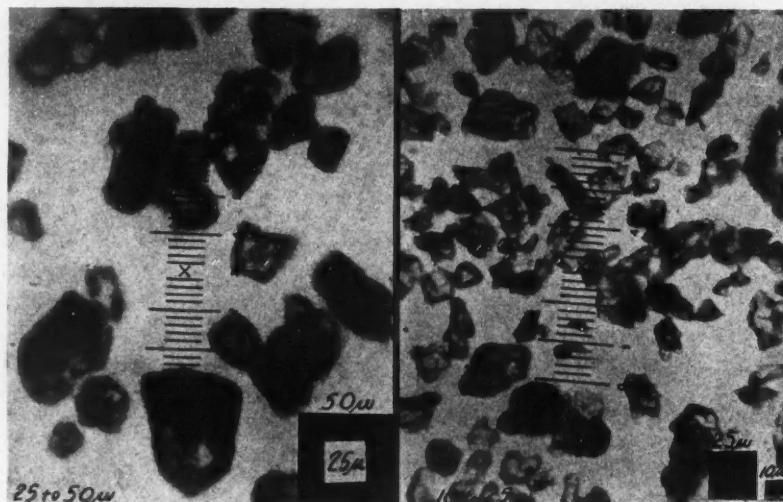


Fig. 4.—Left: 25-micron to 50-micron material; cement of ordinary fineness contains 20 to 30 per cent. of this grading. Right: 10-micron to 25-micron material; cement of ordinary fineness contains 25 to 30 per cent. of this grading.

TABLE I—TEST OF SEPARATED FRACTIONS FOR  $\text{SO}_3$ .  
Original cement: 1.48 per cent.  $\text{S.O}_3$

Grading	Quantity per cent.	$\text{SO}_3$ in contrib. by fraction	each fraction per cent.	$\text{SO}_3$ per cent.
Residue on 200-mesh (over $74\mu$ )	11.2	0.27	0.03	
$25-74\mu$	43.0	0.30	0.13	
$10-25\mu$	23.2	1.01	0.23	
Under $10\mu$	21.6	4.60	0.99	
Dust in filter bag	1.0	10.70	0.11	
Total	..	..	..	1.49 per cent. $\text{SO}_3$

It will be noted that the material coarser than  $25\mu$  contains only  $\text{SO}_3$  derived from the clinker. Practically all the gypsum is concentrated in the material finer than  $10\mu$ . The very fine dust in the filter bag contained 10.7 per cent.  $\text{SO}_3$ , or about 25 per cent. gypsum. The uneven distribution of gypsum in the air-separated fractions makes it impossible to use these fractions for comparative strength tests. Such tests must be carried out on clinker which is ground without the addition of gypsum. Following the air-separation, the individual fractions are then mixed with an equal amount of very fine air-separated gypsum. Only in this way is it possible to obtain comparable results.

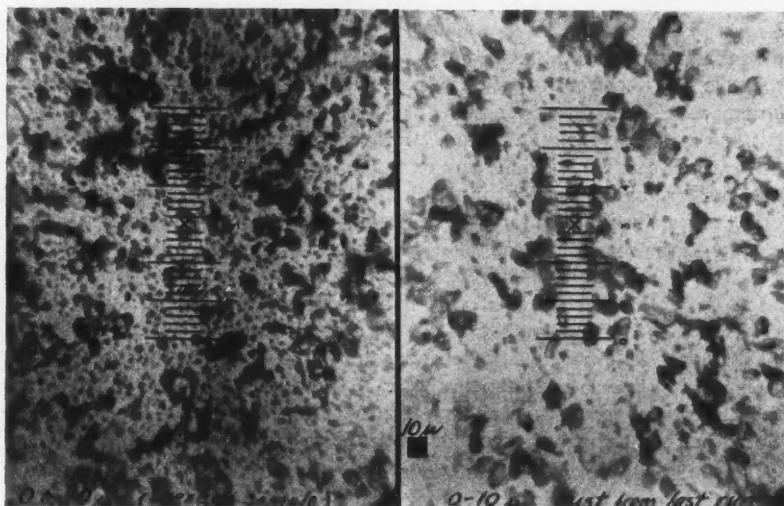


Fig. 5.—Left: Average sample of 0- to 10-micron fraction. Right: Dust recovered from final run of separator of material finer than 10 microns; cement of ordinary fineness contains 20 to 25 per cent. of this grading.

The following example shows the importance of having the gypsum of a sufficient fineness. When it came to determining the amount of gypsum required for regulating the setting time of the cement fraction graded  $0-10\mu$ , a small quantity of seemingly fine gypsum was prepared by a quick shaking of some ground gypsum through a 325-mesh sieve. This sifted gypsum was added to a batch of the fine cement in the proportion of 4 per cent. and the two materials were mixed carefully by being brushed several times through a 200-mesh sieve. A 50-gram pat was made, using 41 per cent. water for obtaining a consistency resembling the normal consistency of regular cement. The pat had a typical "grab set." The initial set (with Gilmore needle) was practically completed

after four minutes, but it was not quite completed before 14 minutes. At 17 minutes the pat commenced to heat up, and final set had taken place after 25 minutes. Another portion of the fine cement was then mixed with 4 per cent. of very fine gypsum, which had been air-separated so that at least 80 per cent. of it was finer than  $15\mu$ . A pat of this cement was made, using the same amount of water as in the previous test. This cement behaved quite normally. It had an initial setting time of one hour, and a final set of six hours. No abnormal heating was noticed. This shows how important it is that the gypsum be sufficiently fine.

#### Main Separation and Tests.

For the main separation tests to determine the range of grading which has the essential concreting value, a batch of clinker from a rotary kiln was ground in a laboratory mill to a fineness of 7 per cent. retained on the 200-mesh sieve. About 40 lb. (18,000 grams) of this cement were air-separated into four fractions, the gradation being: 14.7 per cent., plus  $50\mu$ ; 31.2 per cent.,  $25-50\mu$ ; 29.1 per cent.,  $10-25\mu$ ; 25.0 per cent.,  $0-10\mu$ . The separations were carried out by using 500 grams of cement at a time, and repeating the passing of the cement through the separator until practically constant weight of rejects was obtained. In general it took about thirty passes through the separator to reach constant weight. The progress of the separation was followed closely with the microscope. The total of dust losses for the whole series of separations amounted to 277 grams out of the 18,000 grams treated, equal to a total loss of 1.5 per cent. A close check was kept on the progressive increase of the losses, and as it was evident that the lost material was evenly distributed over the whole range of gradations, it was disregarded in calculating the percentages of the various fractions. Some 2,000 separations were necessary to obtain these four fractions. The average dust loss during each run through the separator is therefore less than 0.15 gram.

The original cement and the four air-separated fractions were tested for chemical composition, with the results given in Table II. It is interesting to study the gradual changes in the composition of the fractions. The coarsest material, representing the material hardest to grind, contains less lime and more silica than the finer fractions. The outer part of the clinker nodules, contaminated with absorbed coal ash and residual sand grains in the clinker, are presumably hardest to grind. The centre of the clinker, which is less contaminated and therefore higher in lime, is softer and easier to grind.

#### Physical Tests.

The four separated cement fractions, and also the original cement, were all given an addition of 4 per cent. gypsum. The gypsum was air-separated so that all of it was finer than  $25\mu$ , and it was judged that about 80 per cent. of it was finer than  $15\mu$ . The amount of  $SO_3$  in the ensuing cements was 1.6 per cent. throughout. The samples were tested for setting time, soundness on boiling, and tensile strength of 1:3 Ottawa standard sand mortar according to the A.S.T.M. specification for testing Portland cement (Table III). In addition, a

series of 3-in. by 6-in. concrete cylinders was also prepared. For the concrete tests a uniform water-cement ratio was maintained, amounting to  $7\frac{1}{2}$  U.S. gallons of water per bag of cement (94 lb.). Based on the weight of dry cement, the amount of water used was 66.5 per cent. The original cement required 24 per cent. of water for normal consistency of the neat cement paste. The fraction graded  $0-10\mu$  required 41 per cent. water for a similar consistency. The paste of this fine cement was extremely fat and sticky. The fractions coarser than  $10\mu$  were all made up with the same amount of water as used for the original cement. When mixed with water the three cements coarser than  $10\mu$  lacked plasticity. Under the trowel they worked like fine wet sand. While being mixed the paste of these cements would seem rather dry and crumbly, but when the paste was formed into a pat on a glass plate the pat would go quite limp and after a short while a large amount of water would exude. A similar feature was observed in the mortars and concretes made with these cements.

The two cements of grain sizes  $25-50\mu$  and coarser than  $50\mu$  had no definite setting times. After about three days storage in a moist closet a carbonated surface film commenced to form on top of the pats. After two weeks storage in the moist closet a surface crust had formed, but underneath this crust the material was still wet and crumbly. After a sufficiently long period the material coarser than  $25\mu$  will develop a certain bonding strength, but it contributes next to nothing to the strength of the concrete during the first month following the placing of the concrete.

The cement fraction graded  $10-25\mu$  had a fairly well-defined setting time, but lacked plasticity to a marked degree. It will be seen later that this cement produced a harsh concrete having a high water segregation.

The fraction graded  $0-10\mu$  had a well-defined setting time. The cement paste had the consistency of butter. The neat paste of this fine fraction developed

TABLE II.—CHEMICAL COMPOSITIONS OF FRACTIONS.

	Original cement per cent.	Fractions			
		Over $50\mu$	$25-50\mu$	$10-25\mu$	$0-10\mu$
$\text{SiO}_2$	...	21.5	22.7	22.3	22.1
$\text{Al}_2\text{O}_3$	...	5.7	6.0	5.6	5.5
$\text{Fe}_2\text{O}_3$	...	2.5	2.7	2.5	2.5
$\text{CaO}$	...	64.6	63.9	64.8	65.4
$\text{MgO}$	...	3.6	3.7	3.8	3.5
Loss on ignition	...	1.9	0.4	0.4	0.7
Calculated on no loss on ignition basis the analysis would be:					
$\text{SiO}_2$	...	21.9	22.8	22.4	22.3
$\text{Al}_2\text{O}_3$	...	5.8	6.1	5.6	5.5
$\text{Fe}_2\text{O}_3$	...	2.6	2.7	2.6	2.5
$\text{CaO}$	...	65.9	64.2	65.0	65.9
$\text{MgO}$	...	3.7	3.7	3.8	3.5

TABLE III.—TESTS ACCORDING TO A.S.T.M. SPECIFICATIONS

		Original cement	Fraction 10-25 $\mu$	Fraction 0-10 $\mu$
Initial setting time	.. ..	3:00	7:00	1:00
Final setting time (Gilmore needle)	.. ..	4:40	11:00	6:00
Boiling test	.. ..	Sound	Sound	Sound
Tensile strength (1:3 Ottawa sand briquettes stored 1 day in moist air then in water of 70 deg. F.)				
Water used, in % of dry mortar	10.5	10.5	13.4	
1 day	.. ..	121 lb. sq. in.	40 lb. sq. in.	218 lb. sq. in.
3 days	.. ..	227	118	420
7 days	.. ..	311	294	443
28 days	.. ..	432	450	478
90 days	.. ..	459	515	481
1 year	.. ..	338	428	440

shrinkage cracks before final set had taken place, although the cement pat was kept in an atmosphere of 100 per cent. humidity. No unusual shrinkage was noted when the fine cement was used in concrete, but it is certain that a cement of this type will produce a higher initial shrinkage of concrete than would cement of usual fineness. The fresh concrete made with this fine cement was very plastic and workable, and there was no trace of water segregation. About five minutes after being made this concrete had a considerable initial stiffening as the water was absorbed by the swelling cement particles, but no unusual heating was observed.

Three briquettes were tested for each period. No briquettes were made of the two coarsest fractions, as the water segregation from the mortar was judged to render the ensuing test results unfit for comparisons. The retrogression in strength of the one-year-old briquettes is not indicative of a true deterioration, as it is a fairly common phenomenon with briquettes older than ninety days.

Judged by these values for tensile strengths there should be no very great difference in the strengths of the three cements. Even the very fine cement is not much ahead of the original cement. This agrees with the conclusions arrived at by Kühl, but we get an entirely different picture when the cements are tested in concrete (Table IV).

TABLE IV.—TESTS IN CONCRETE.

Compressive strengths of concrete in lb. per sq. in. Water-cement ratio equal to  $7\frac{1}{2}$  U.S. gal. per 94 lb. cement (66.5 per cent. water by weight of cement). 3-in. by 6-in. cylinders, cured in moist air at 70 deg. F. Two cylinders tested at each period.

	Age of cylinder	Original cement	Fraction 10-25 $\mu$	Fraction 0-10 $\mu$
1 day	.. ..	234	206	1000
3 days	.. ..	1047	906	3430
7 "	.. ..	1948	1965	4000
28 "	.. ..	2960	3410	4200
90 "	.. ..	3950	4140	4530
1 year	.. ..	5080	5110	5450

The aggregates used for the concrete mixes were clean, well-graded river sand and graded  $\frac{1}{4}$  in. to  $\frac{3}{4}$  in. gravel. The proportioning of the mixes is given on a volume basis to conform with usual practice, but the actual proportioning was done by weighing the materials. In adding water to the concrete mixes allowance was made for the water absorbed by the room-dry aggregates. The cement weighed 94 lb. per cu. ft., the sand 103 lb. per cu. ft., and the gravel 100 lb. per cu. ft. The weight per cu. ft. of the different cement fractions was assumed to be the same (94 lb.). In practice there is of course a very considerable difference in the bulking of the various fractions.

#### Discussion of Concrete Mixes.

**ORIGINAL CEMENT.**—The concrete made with the original cement was proportioned 1:2.5:3.3 by dry rodded volumes. The slump of this concrete was  $1\frac{1}{2}$  in. and the workability excellent. Water segregation from the mix was almost nil.

**CEMENT 0-10 $\mu$ .**—This fine cement was first tried in a small batch of concrete having the same composition as the mix for the original cement. But as this mix was very stiff and difficult to work the ratio between the sand and gravel was altered so that the final mix contained less sand and more gravel. The mix from which the concrete cylinders were made up was proportioned 1:2.2:3.6 by dry rodded volumes. The slump was about 1 in. and the plastic workability of the fresh mix was very good. After about five minutes an initial stiffening was observed. The concrete had been placed in the test cylinders when this stiffening took place so it did not interfere with the quality of the concrete cylinders, but in practice this stiffening would have caused difficulty.

**CEMENT 10-25 $\mu$ .**—The cement graded 10-25 $\mu$  was first tried in a concrete mix of the same proportions as was used for the original cement, but the ensuing concrete was very harsh and quite "sloppy." There was no coherence between coarse and fine aggregate. More sand was added to improve the workability of the mix. The final mix with this cement was oversanded judged by ordinary standards, but it was found necessary to use an excess of sand to reduce the water segregation. The concrete from which the test cylinders were prepared was proportioned 1:2.8:3.3 by dry rodded volumes. The slump was about 3 in. It was a concrete of poor workability, and under practical working conditions it would have been very difficult, if not impossible, to place without the formation of stone pockets and honeycombed patches. In spite of all precautions a considerable amount of water exuded from the mix.

**CEMENTS COARSER THAN 25 $\mu$ .**—The two fractions coarser than 25 $\mu$  were tried in concrete mixes, but the mixes were not much different from a plain wet mixture of sand and gravel. The water segregation was so excessive that it would have been impossible to obtain strength tests of any value. For this reason no specific tests were made to determine the bonding strength of the material coarser than 25 $\mu$ . During the first several weeks' life of the concrete this coarse material is nothing but an inert filler. At later ages the coarse grains contribute in a small way to the compressive strength of the concrete.

It is the material finer than 10 $\mu$  which, to begin with, must carry the dead

weight of the coarser material, and it is this fine flour which is responsible for the plastic workability of the concrete mix, without which quality it would not be possible to make an impervious and durable concrete. The early hardening power of the concrete is likewise determined by the material finer than  $10\mu$ . At the age of seven days this material has developed a concrete strength which is about 78 per cent. of the strength developed by the original cement after one year. If strength alone were considered one might say that the material finer than  $25\mu$  represented the flour in the cement. But it is the material finer than  $10\mu$  which alone contains the two essential qualities of strength and plasticity. It is this material which forms the "back-bone" of the cement, and should be termed the essential flour.

#### Tests with Mixed Cements.

In addition to the tests on the separated fractions of the cement, two series of tests were made on cements mixed to a predetermined grading. The object of these tests was to gain an idea of the quality improvement which might be

TABLE V.—GRANULOMETRIC COMPOSITIONS OF CEMENTS.

	Original cement.	Cement A.	Cement B.	High-early strength cement.
	per cent.	per cent.	per cent.	per cent.
Fineness on 200-mesh (amount retained)	7.0	0	0	3.2
Granulometric composition:				
Over $50\mu$	14.7	nil	nil	not determined
$25-50\mu$	31.2	42	nil	..
$10-25\mu$	29.1	36	60	..
$0-10\mu$	25.0	22	40	45

TABLE VI.—TESTS OF MIXED CEMENTS IN CONCRETE.

Compressive strengths of concretes in lb. per sq. in.

Water-cement ratio equal to  $7\frac{1}{2}$  gall. per 94 lb. cement (66.5 per cent. water by weight of dry cement).

Age of cylinder.	Original cement.	Cement A.	Cement B.	High-early strength cement.
1 day	234	262	474	1025
3 days	1047	1228	2080	2100
7	1948	2050	3000	2400
28	2960	3200	3950	3000
90	3950	—	—	3200

expected by a perfected air-separation on a large scale. The granulometric compositions of these two mixed cements and of the original cement, and a partial analysis of a commercial high-early strength cement are given in Table V. Cement A is supposed to represent an air-separated cement having only a trace of residue on the 325-mesh sieve. To obtain such a cement it would be necessary to reject and regrind 20 to 25 per cent. of a cement ground to ordinary fineness. Cement B is one in which everything coarser than  $25\mu$  has been removed. This would mean that about 50 per cent. of an ordinary cement

would have to be rejected and reground. In proportioning these two mixtures due consideration has been given to the fact that a closed-circuit grinding system, consisting of a mill of the present-day type coupled to a single-action cyclonic separator, will produce less of the very finest material than one would expect by simply calculating the probable composition of the separated cement on the basis of the granulometric composition of the cement produced when the mill is grinding without air-separator.

Both of the mixed cements were given an addition of 4 per cent. air-separated gypsum. The setting times of both cements were quite normal, and about the same as that of the original cement. The available amount of the two mixed cements was only sufficient for concrete cylinders up to an age of twenty-eight days. No briquettes for tensile tests were made. Both cements were tested in concrete mixes having the same water-cement ratio as was used for previous concrete tests. The proportioning was 1:2.6:3.6 by dry rodded volumes. Cement A had practically the same characteristics as the original cement. Concrete made with Cement B had a very superior plasticity; it was judged to be even better than that of the concrete made with the finely ground high-early strength cement. Cement B did not show any objectionable initial stiffening.

These tests seem to indicate that a very considerable amount of air-separation and regrinding would be required to produce any marked increase in concrete strength and might also indicate that air-separation should not be looked upon as a very efficient means towards strength improvement. But when air-separation is applied correctly it should show considerable saving in the cost of the grinding operation.

In the March 1932 issue of *CEMENT AND CEMENT MANUFACTURE* is published a paper by Helbig, "Multi-chamber Mills with Air Separation for Superfine Cement." The writer considers this a valuable and constructive discussion of the present-day problems of cement grinding. Helbig advocates a system of cement grinding consisting of a preliminary grinder in closed-circuit with a vibrating screen and a finishing mill in closed-circuit with an air-separator. When correctly designed such a system should offer a solution to many of the present-day grinding difficulties.

Air-separators in closed-circuit with finishing mills for cement have been in use for several years, but opinions differ somewhat as to the merits of this system. It would seem that much of the criticism against this system is based on unsatisfactory results obtained with installations not designed to take full advantage of the inherent possibilities of closed-circuit grinding. Several of the air-separators in use for cement grinding have been installed as an afterthought and are coupled to grinding mills designed for open-circuit grinding. Such a mill will not have maximum efficiency when handling the much larger load used in the closed-circuit system. Another drawback to this method of grinding is the relatively low efficiency of the single-action cyclonic separator. The problems of an efficient air-separation which would produce two fractions, one containing, say, 95 per cent. of all material finer than  $25\mu$ , and the other 95

per cent. of all coarser material, are very similar to the problems of fractionated distillation of two liquids having boiling points close together (alcohol-water). The efficient separation by distillation of alcohol and water is only possible by using a multiple action distillation column. In a similar manner the efficient air-separation of cement undoubtedly depends on the design of a multiple-stage separator. The cement would flow through this separator only once, but while passing through it would be exposed to numerous separation stages, each mild in action and removing only a part of the desired fine material. The total of the work performed would be almost quantitative separation.

In concluding, I wish to express my appreciation of the help given me by Mr. A. T. Harwood, chief chemist of the Florida Portland Cement Co., who has conducted all chemical tests, and Mr. J. E. Tonetti, of the Signal Mountain Portland Cement Co., who has assisted me with the air separations.

[A comment on this article, from the United States Bureau of Standards, will be given in our next issue.]

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## Notes from Abroad

### United States Cement Companies' Reports.

Lehigh Portland Cement Co. reports for the twelve months ended September 30, 1932 a net loss of 1,329,328 dollars (£273,524). This compares with a net profit of 482,352 dollars (£99,249) for the twelve months ended September 30, 1931.

Pennsylvania-Dixie Portland Cement Co., for the twelve months ended June 30, 1932, showed a net loss of 1,643,140 dollars (£338,094). In the preceding year the loss was 115,551 dollars (£23,775).

The Alpha Portland Cement Company closed its financial year at June 30, 1932, with a net loss of 1,193,449 dollars (£245,565), against a net profit of 546,583 dollars (£112,465) in the previous year.

### Belgian Cement Company's Report.

S. A. des Ciments Meuse-Bragant has passed its dividend for the year ended September 30, 1932. For the previous year a dividend of 10 per cent. was paid.

### French Cement Company's Report.

Soc. Mediterranienne des Chaux et Ciments Portland Artificiels report a net profit of Frs. 301,015 (£2,423) for the year 1931, and a gross dividend of Frs. 30 (4s. 10d.) per share has been declared.

### Belgian Congo.

The net profit of Société des Ciments du Congo for the year 1931 was £21,670, compared with £25,082 in 1930 and £8,244 in 1929. No dividends were paid for either of these years. During the past year the company has completed the installation of a third kiln at Lukala, which will enable the works to produce 40,000 tons of artificial Portland cement per annum. According to the *Bulletin de l'Office*, cement production in the Congo-Kasai Province during 1931 amounted to 22,287 tons, compared with 23,296 tons for the preceding year.

## Facility of Burning Portland Cement.

At the meeting of the Association of Japanese Portland Cement Engineers, held in Tokio in November last, Mr. Keiichi Akiyama, of Waseda University, read a paper entitled "The Facility of Burning Portland Cement depends on its Hydraulic Modulus, Silica Ratio and Iron Modulus," of which the following is an abstract.

Synthetical raw material consisting of pure chemicals of Kahlbaum was well mixed in the following proportion, kneaded with water into small balls, dried, burnt in the electric furnace in platinum vessels and rapidly cooled.

$\text{SiO}_2$	24	Hydraulic Modulus	$\frac{\text{CaO}}{\text{SiO}_2 + \text{R}_2\text{O}_3} = 2.00$
$\text{Al}_2\text{O}_3$	6		
$\text{Fe}_2\text{O}_3$	3	Silica Ratio	$\frac{\text{SiO}_2}{\text{Al}_2\text{O}_3} = 2.67$
$\text{CaO}$	66		
$\text{MgO}$	1	Iron Modulus	$\frac{\text{Al}_2\text{O}_3}{\text{Fe}_2\text{O}_3} = 2.00$
	100		

The temperature was kept for 30 minutes at 1,150, 1,250, 1,300, 1,350, 1,450 deg. C. respectively. The burnt product was, after pulverisation, tested for free CaO by Lerch and Bogue's method. The result is shown in Fig. 1.

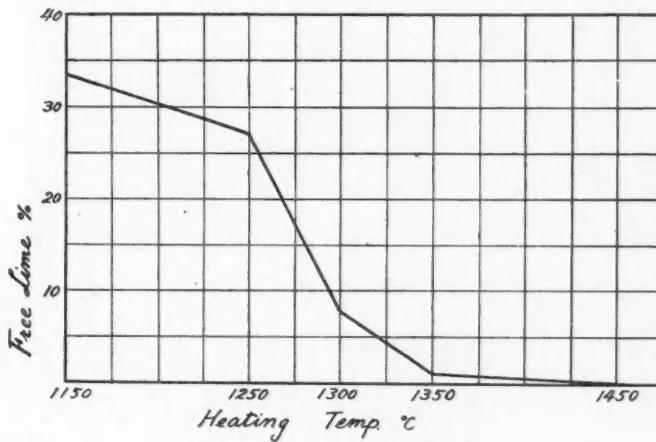


Fig. 1.

The conclusion reached is that although the temperature of perfect sintering is 1,450 deg. C., for the purpose of studying the facility of lime combination 1,350 deg. C. is suitable.

In another experiment, 30 raw mixtures of different proportions were prepared with hydraulic moduli of 2.0 and 2.2, silica ratios of 3.3, 3.0, 2.7, 2.4, and 2.1, and iron moduli of 3.0, 2.0 and 1.5 heated in an electric furnace at 1,350 deg. C. for ten minutes and cooled rapidly. The free CaO contents of the burnt cement are shown in Fig. 2, which is self-explanatory. The average of free CaO in the upper three lines is nearly three times larger than the lower three, which shows that the burning of high-limed cement is very difficult. On the vertical line of  $\frac{\text{SiO}_2}{\text{Al}_2\text{O}_3} = 3.3$  the average of free CaO content is approximately 10.7 per cent. and 3.8 per cent. for hydraulic moduli of 2.2 and 2.0 respectively

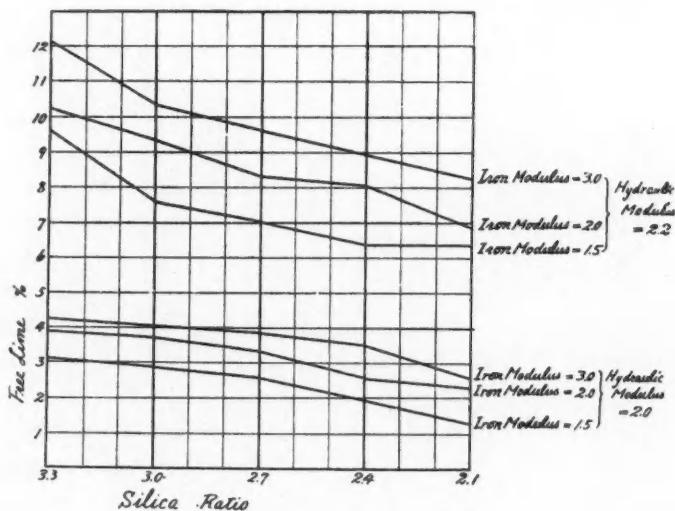


Fig. 2.

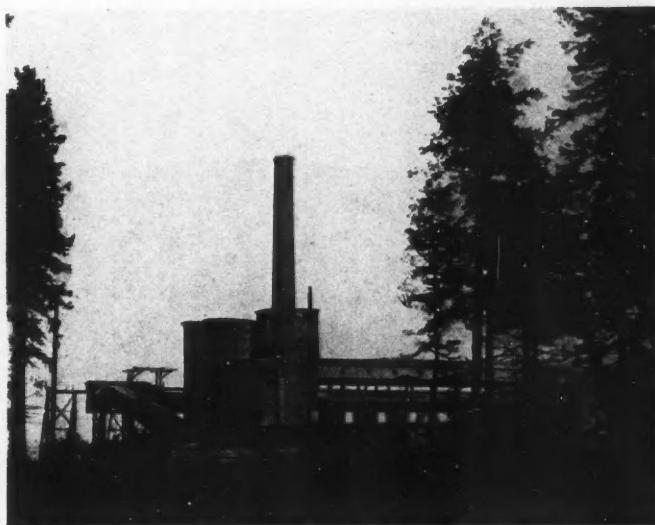
whereas free CaO corresponding to  $\frac{\text{SiO}_2}{\text{Al}_2\text{O}_3} = 2.1$  is 7.2 per cent. and 2.0 per cent.

The decrease of  $\frac{\text{SiO}_2}{\text{Al}_2\text{O}_3}$  facilitates the fusion, the influence being more marked in the high-limed cement. The variation of iron modulus  $\frac{\text{Al}_2\text{O}_3}{\text{Fe}_2\text{O}_3}$  from 3.0 to 1.5 reduces the free-lime content a little more than 1 per cent. in low-limed cement and more than 2 per cent. in average in high-limed cement. Therefore the influence of iron modulus on the facility of fusion is more marked the higher the hydraulic modulus.

The conclusion is reached that the proper selection of silica ratio and iron modulus is of great importance in the manufacture of high-limed cement.

## New Cement Works in Sweden.

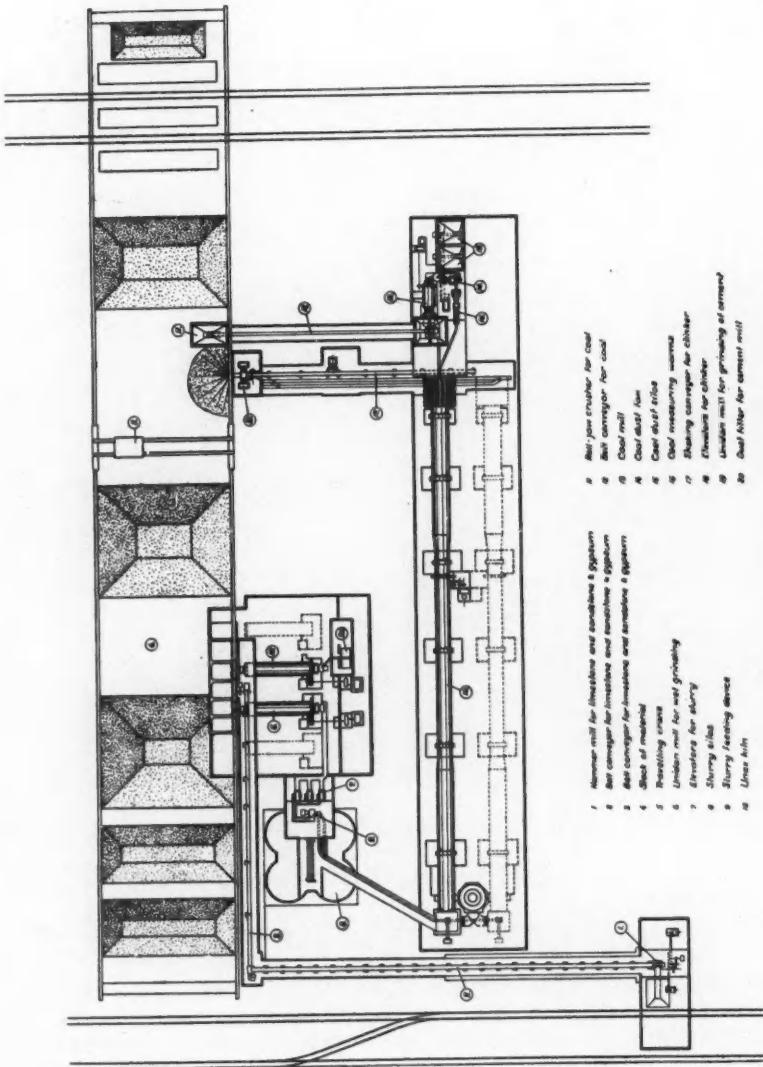
THE endeavours of cement manufacturers to improve the quality of their product and at the same time to reduce prices, have caused the manufacturing costs of many old and out-of-date cement works to be out of touch with current cement prices. In certain cases a thorough reconstruction and modernising of old plants have resulted in economies that have enabled them to compete successfully. In other cases the erection of entirely new works is preferred to the reconstruction of old ones. After such consideration the new cement works described in this article was erected in the years 1929 to 1931 by Skånska Cement Aktiebolaget at Hellekis in Sweden. This works is situated at the foot of Mount Kinnekulle,



New Cement Works at Hellekis, Sweden.

on the southern side of Lake Wener, a few hours by rail from Gothenburg in the direction of Stockholm.

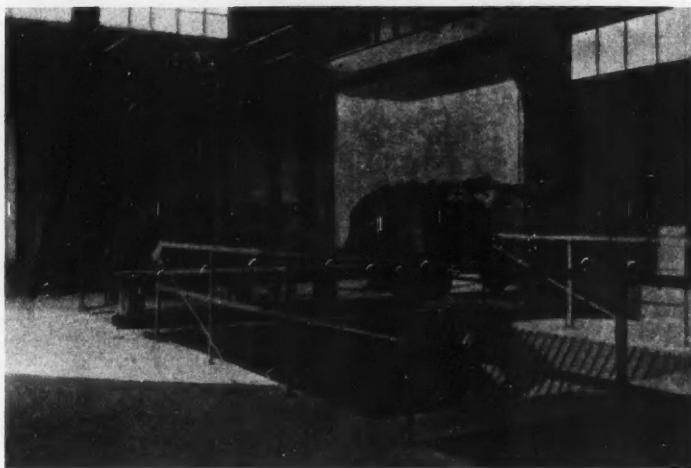
The older works on the same site was started in 1892 with a capacity of 7,500 barrels of cement a year. In 1906 and 1907 the works was equipped with two 100ft. rotary kilns. In 1928 the factory had reached a capacity of about 340,000 barrels of cement a year. The plant, which worked on the dry process, was equipped with four crushers, thirteen grinding units for raw meal, two rotary kilns, two shaft kilns, and five cement mills; it was thus very complicated. As the demands for better and cheaper cement became more and more insistent it became obvious that a new plant was needed, and in 1929 the company applied to F. L.



Plan of New Cement Works at Hellekis, Sweden.

Smidh & Co. A/S., of Copenhagen, to design an extension of the factory. Several methods of manufacture were taken into account in the preliminary investigations: particularly whether the dry or the wet process should be employed, whether cement burning should be effected in shaft kilns or in rotary kilns, whether waste heat from the rotary kilns should be utilised in waste-heat boilers, or whether a highly economical rotary kiln would be preferable. The rotary kiln type was finally chosen in connection with the wet process as the most economical, the simplest, and the best plant from the point of view of quality.

The new plant is capable of producing 500,000 barrels of cement a year, a raw grinding department, kiln department, and cement grinding department being provided with main machinery for this capacity. The buildings for these depart-



Crushing Department.

ments have, however, been so designed that there is room for an extension to double the present capacity, each department providing accommodation for another unit of the same size as the existing one. Other devices, such as transporters, crushing installation, coal mill, power mains, transformers, etc., have already been built for double the present capacity.

In each of the main departments there is only one unit of machinery, namely, one crusher, one raw mill, one rotary kiln, one coal mill, and one cement mill, so that the process of manufacture is as simple as possible. With regard to reliability of working, it is notable that only six of the forty-seven electric motors in the factory are provided with belt drives; the others are either coupled directly to the working machines, or a gear is arranged between motor and working machine. To further ensure smooth working in case one of the main machines should fail for a short time, considerable stocks of raw materials as well as of semi-

manufactured and finished products are allowed for. Thus it is possible to store at the same time 12,000 tons of crushed limestone, about 1,000 tons of gypsum, about 100,000 cu. ft. of raw slurry, about 17,000 tons of clinker, about 4,000 tons of coal, and about 50,000 barrels of cement. The requirement of manual labour has been reduced to a minimum. The plant works with three shifts per twenty-four hours, with only ten men in each shift besides some day-workers such as repairers, transport workers, and quarry-men.

The raw materials consist mainly of two different kinds of limestone : orthoceratite limestone of Cambrian-Silurian formation, and a sandstone. Most of the limestone is quarried in an open quarry about two miles from the works. The face of the quarry is about 2,500ft. long and 40ft. high. The sandstone, of which



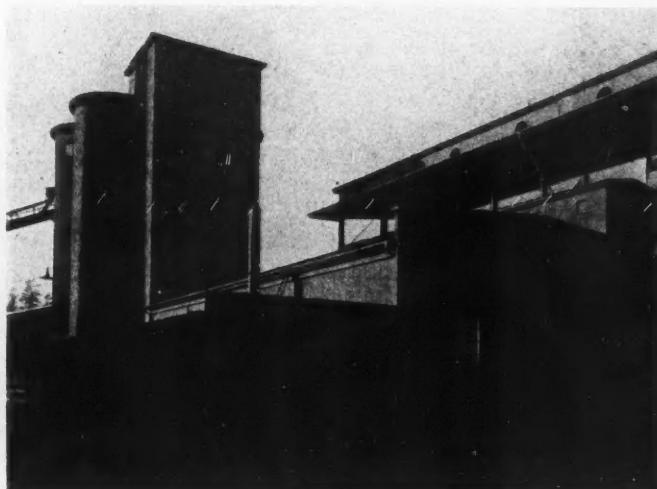
Raw Material Store, Slurry Silos and Kiln House.

only a small amount is required, is quarried in the immediate neighbourhood of the factory.

#### Crushing Department and Raw Mill.

The stone is transported from the quarry to the crushing department in railway trucks, which are shunted on to a tipping platform arranged as a wagon weigher, and from which the material, after having been weighed, is thrown down into a hopper above a horizontal laminated feeding band 16ft. long driven by a variable-speed motor. On this band the material is carried to a slow-speed double hammer crusher, the two shafts of which are driven separately, each by a 100 h.p. motor. The crusher can cope with limestone pieces up to 2ft. 4in. by 2ft. 4in. by 1ft. 8in. and crush the material down to a maximum grain size of about 1in., with about 50 per cent. below 0.4in. The crusher will normally crush about seventy tons per hour, and the dust produced by crushing is removed by a special dust-collecting plant.

From the hammer mill the crushed material is taken by a 250ft. long inclined rubber belt conveyor up to a horizontal steel belt conveyor 165ft. long running along the raw material store at such a height that the crushed raw materials can either be distributed to various points of the raw material store or be carried to the hoppers above the raw mills. This store is arranged as a covered crane store parallel with and immediately beside the mill and kiln houses. The store is served by a 7.5-ton four-motor electric travelling crane with grab. The crane bridge has a span of 80ft. and a maximum height of lift of 50ft. As this crane performs a number of duties (unloading coal, moving clinker, gypsum, limestone, etc.), it is of vital importance for the working of the factory and is therefore provided with a complete set of spare motors.



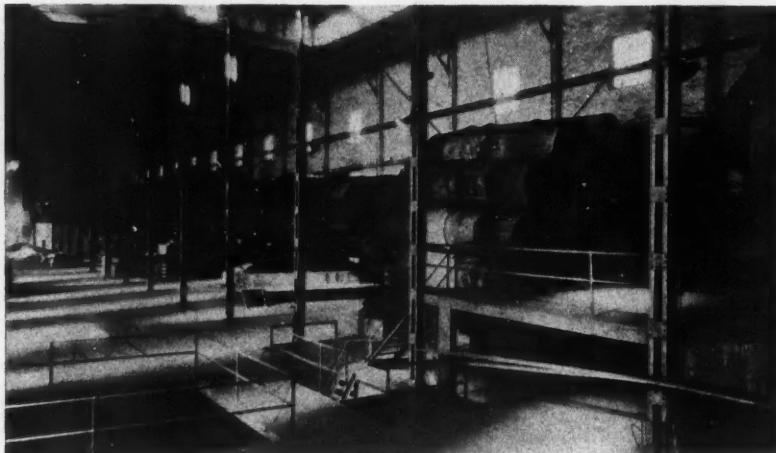
**Grinding Department, with Slurry Silos in the Background.**

Above the raw mill there are three hoppers, two for limestone and one for sandstone. From the two former hoppers limestone is fed to the mill by means of two feed tables having a common countershaft direct coupled to a variable-speed motor, whereas the sandstone from the third hopper is supplied by means of a push feeder. The raw mill is a Unidan mill for wet grinding, 40ft. long, with three chambers, and driven by a 650 h.p. motor. The normal output of the mill is about twenty tons of dry material per hour to an average fineness of 10 per cent. residue of the 4,900 mesh sieve.

From the raw mill the ground slurry runs in a trough to a small collecting pit serving three slurry elevators, which are erected in reinforced concrete casings about 100 ft. high and have a transporting capacity of about 900 cu. ft. per hour

each. One elevator takes the slurry from the mill to the slurry silos ; the second takes the slurry from the silos to the kiln ; whereas the third is kept as spare and can also be used if necessary for correction of the slurry.

The slurry is stored in four silos which are more than 65 ft. high and have a total capacity of about 100,000 cu. ft. The silos are used partly for storing the mixed kiln slurry and partly as correction basins. Usually the correction is effected by varying the feed of the various materials. In order to homogenise the slurry in the silos, compressed air is intermittently blown through valves into the bottoms of the silos. The addition of compressed air is regulated automatically and takes place by means of two compressors installed in the kiln house, each having a capacity of 530 cu. ft. of intaken air per minute. One of the compressors is kept as spare.



The Kiln.

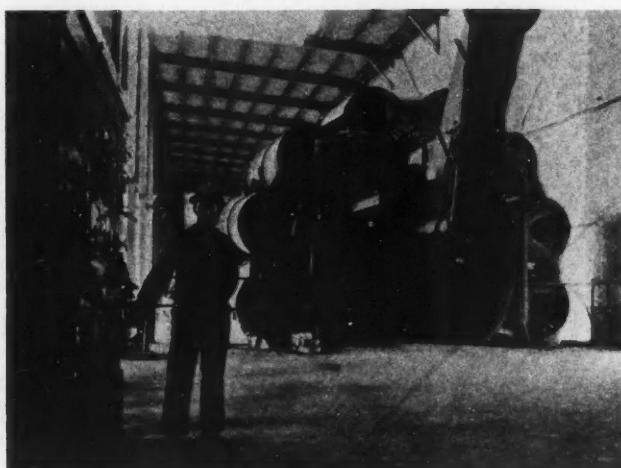
#### Kiln Department.

The slurry is fed to the kiln by means of a rotary slurry feeder driven by a variable speed direct-current motor. A light and sound indicator is acted upon by the current of slurry fed to the kiln and warns the burner of irregularities in the slurry supply. The rotary kiln is a 310 ft. Unax kiln, at the outlet end of which twelve cooler tubes are fitted and move concentrically with the kiln. The diameter of the kiln shell is smallest at the middle, the kiln being provided at its lower end with a widened burning and calcining zone and at its upper end with a widened slurry drying zone with chain installations for improving the heat transfer. The normal kiln output per twenty-four hours is about 260 tons of clinker with a fuel consumption of less than 23 per cent. of standard coal of 12,500 B.T.U. calorific value. The effective cooling of the clinker, which leaves

the cooler at about 200 deg. F., contributes to the achievement of such good heat economy. With the exception of a certain amount of hot air which is taken out for drying the coal, all the clinker heat is utilised, according to the design of the Unax kiln, for preheating the air for combustion. The kiln draught is produced by a fan, but, by changing over to a by-pass, natural draught can be used. At the bottom of the smoke chamber the dust is discharged automatically by means of a drag chain; an elevator lifts the dust up to the slurry chute of the kiln and returns it to the kiln together with the slurry.

#### Coal Mill.

Coal is delivered in railway trucks at one end of the raw material store, whence it is distributed in the coal store by means of the grab of the travelling

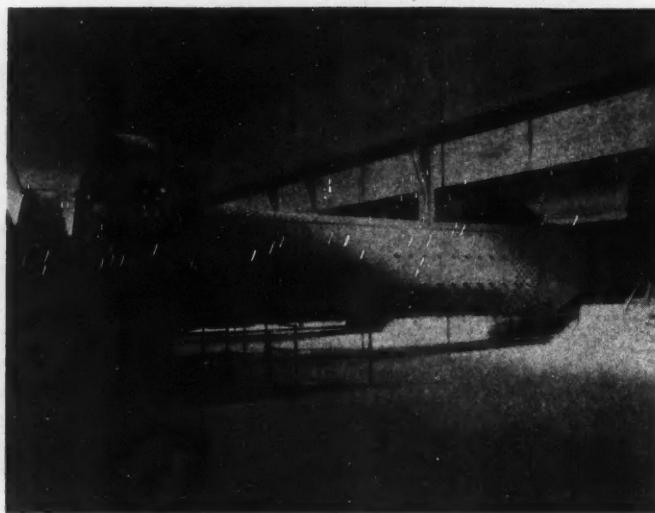


**Burner's Platform, with Instrument Board in the Foreground.**

crane. From the store the coal is taken to the hoppers above the roll-jaw coal crusher; these hoppers also act as a feeding device for the 150 ft. inclined steel belt conveyor which carries the coal to the hopper above the coal mill.

The coal mill is a 13 ft. long tube mill which is ventilated with hot air, and which at its inlet end is provided with a special drying chamber with lifting and distributing devices through which the hot air passes and in which some of the water is dried off the coal. From this chamber the coal is led through a hollow trunnion into the first chamber of the mill and then through a diaphragm into the second chamber. The hot-air circulation is effected by a high-pressure fan driven by a 50 h.p. motor and by which a current of preheated air is sucked through the mill from the lower end of the Unax kiln. This current of air carries away the ground coal, which is separated in a cyclone the efficiency of which

has been measured as 95.5 per cent. Thence the coal dust is transported by a 43 ft. long worm conveyor and an elevator of about 50 ft. in height, as well as a further worm conveyor, partly to the coal-dust hopper in front of the Unax kiln and partly, so far as the excess coal is concerned, to two coal-dust silos. The combined coal drying mill is designed to supply coal dust to a further Unax kiln of the same dimensions as those of the existing one. Under present conditions, where the mill has only one-half of the amount of air available for the operation, the output is about six tons per hour with 6 per cent. of moisture in the rough coal and 1.6 per cent. in the coal dust, which is ground to a fineness of about 14 per cent. residue on the 4,900-mesh sieve and 0.6 per cent. residue on the 900-mesh sieve. In this preliminary working the 200 h.p. motor of the mill



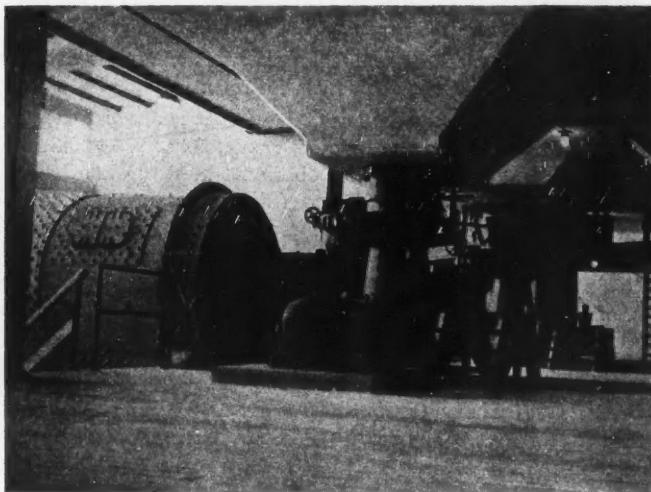
**Mills for Grinding Slurry and Cement (Cement Mills in the Foreground).**

is loaded to only two-thirds of its normal capacity. The coal is measured to the Unax kiln by means of a double coal measuring worm.

From the outlet of the Unax kiln the clinker falls into a 135 ft. long shaking conveyor built in an underground tunnel. At the outlet of the shaking conveyor a grate device is arranged which separates the fine clinker, so that this is taken directly to one of the two clinker elevators built in a concrete casing about 80 ft. high, while the coarser clinker first passes a small stone crusher before it is lifted by the same clinker elevators above the clinker store. At the upper end of the elevators there is an automatic weigher, by which the amount of clinker supplied to the clinker store is checked.

**Cement Mill.**

The cement mill is arranged in the same building as the raw mill, so that both mills can be attended by one man, and, like the raw mill, it is a Unidan mill 40 ft. long, driven by a 650 h.p. motor. Clinker and gypsum are measured to the mill by two rotating feed-tables. The mill grinds about 17 tons of clinker per hour to a fineness of about 7 per cent. residue on the 4,900-mesh sieve. A fan sucks the dust-filled air from the interior of the mill through a dust filter in which the dust is collected in hoses of filter cloth. The amount of cement produced is checked by an automatic weigher directly connected with the mill outlet, whereupon the cement is transported pneumatically by compressed air of about 5 atmospheres through a 4 in. pipeline for a distance of about 650 ft. to the



**Inlet End of the Mills, with Feed Tables.**

cement silos situate at the old plant, where the cement is packed in paper valve sacks.

In a cement works where mechanisation has been carried so far as in this case, and where the number of men required for continuous shift working in the factory has been reduced to a minimum, extensive safety and control arrangements are required. The most important post of operation is, as in any other cement works, the burner's platform. This place is, therefore, equipped with the following control apparatus for assisting the burner:  $\text{CO}_2$  meter and CO meter for measurement of the hot gases in the chimney; temperature meters for measuring the gas temperature at the kiln inlet, and the temperatures in the inlet end and the outlet end of the coal mill; revolution counter for the kiln;

pressure gauge showing under-pressure in smoke chamber, under-pressure in coal mill, and pressure in water pipelines; regulating devices for the coal measuring worm, for the chimney damper, for the kiln motor, and for the slurry-feed motor. There is a telephone connection to the slurry feeding place at the upper end of the kiln. Sound and light signals warn the burner if interruptions in the slurry supply occur, if the exit gases have too high a temperature, if there is too low a pressure in the cooling-water pipelines, too high a temperature in the coal mill, and too high a temperature in the transformer.

The plant was started on June 29, 1931, and the Unax kiln worked continuously from that day until Christmas, 1931, when it was stopped on account of the holidays. The plant is still producing to capacity, the economy of production enabling it to compete successfully. The quality of the cement is excellent.

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#### U.S.A. Cement Companies' Reports and Dividends.

The Bessemer Limestone & Cement Co. show a loss of 875,776 dollars for the year 1931, against a profit of 267,766 dollars for 1930.

The Lehigh Portland Cement Co. has declared the regular quarterly dividend of 1.75 dollars on the preferred stock, payable on October 1. The Secretary of the company stated: "In view of the substantial impairment to surplus and current available resources of the company within the past year it is apparent that improvement in the earnings of the company, which will be possible only in the event of an increased volume of trade, must in the future determine dividend action on the preferred stock." For the year ended June 30, 1932, there was a loss, after provision for depreciation and obsolescence, of 484,020 dollars, while for the second quarter of 1932 there was a loss of 406,386 dollars.

#### French Cement Company's Dividends.

Soc. Générale des Chaux et Ciments reports a net profit of Frs. 842,225 (£6,780) for the year 1931. The board state that production in 1931 was 8½ per cent. less than in 1930.

## BINDING CASES for "Cement & Cement Manufacture."

Strong binding cases for the 1932 volume of "Cement & Cement Manufacture" are now ready, price 3s. 6d. (by post, 3s. 9d.) each. These cases are cloth covered, with the title of the journal and the date of the volume blocked in gold on the side and spine. If desired, we will undertake the work of binding at an inclusive charge of 6s. plus 6d. postage; in this case the twelve numbers should be sent post paid to Concrete Publications, Ltd., 20 Dartmouth Street, London, S.W.1. For the information of those who may wish us to complete their sets, all the 1932 numbers are in print and can be supplied, price 1s. each.

## A Wet-Process Cement Plant.

THE wet-process kiln illustrated has been installed to increase the capacity of an existing cement plant. The additions consist in the main in the form of an oil-fired "Concentra" rotary kiln of 9 ft. to 9 ft. 10 in. diameter by 223 ft. long, with two-compartment tube mills 7 ft. 3 in. in diameter by 42 ft. 8 in. long, with "Centra" drive and the necessary auxiliary equipment, supplied by Krupp-Grusonwerk. In conjunction with the kiln there was also installed a waste-heat steam-raising plant, with turbine to provide the works with electricity.

In regard to the provision for cooling the burnt clinker, the rotary kiln (Fig. 1) involves a new departure. It carries at its discharge end, where the diameter is 9 ft. 10 in., twelve cooling cylinders of 35 in. diameter uniformly spaced around

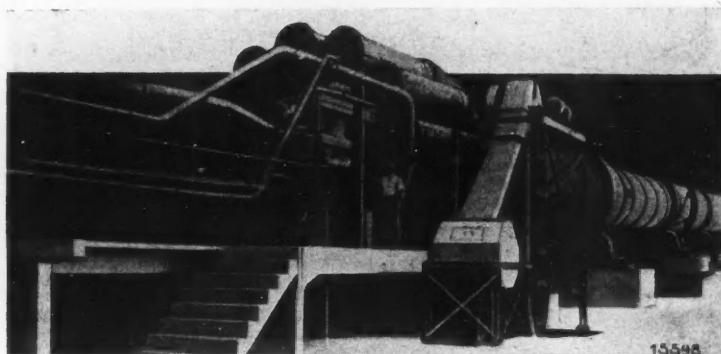


Fig. 1.

its circumference. The arrangement of the cooling cylinders and the method of support result in low headroom requirements and ensure dependable working. By this subdivision of the cooler, separate small cooling chambers are formed so that the combustion air flowing to the kiln through the twelve cooling cylinders comes into such effective contact with the hot clinker that the latter is efficiently cooled and the combustion air enters the kiln at a high temperature. Another source of increased cooling effect lies in the fact that the clinker travels along the cooling cylinders in the opposite direction to its progress in the kiln, and hence it is exposed to the cooling air for a longer period. In Fig. 2 the path of the cooling air (A) and of the clinker (B) in the kiln and in the cooling cylinders are shown. The temperature of the clinker on leaving the cooler, with a daily kiln output of approximately 200 tons, is about 212 deg. F.

The cooling cylinders are protected from wear and radiation losses are minimised by a lining provided with strong scoops for lifting and dropping the clinker.

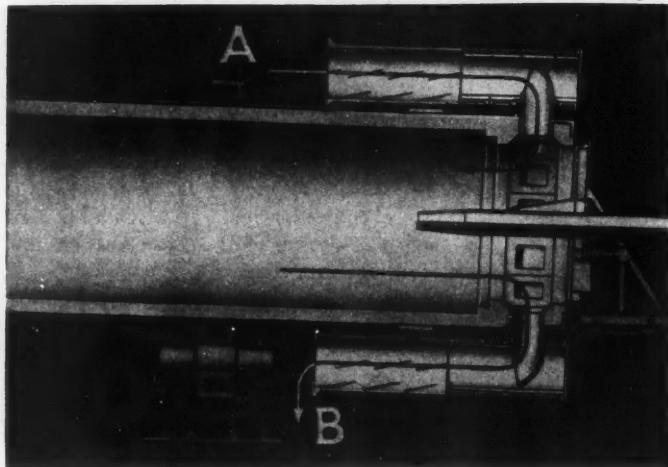


Fig. 2.

At the points where the clinker leaves the kiln for the coolers the clinker is spread out by cooling-pieces fitted in the kiln, and thereby undergoes preliminary cooling. These cooling-pieces are made of special heat-resisting steel which retains great hardness even at high temperatures. Through the cooling cylinders there passes 90 per cent. of the total combustion air.

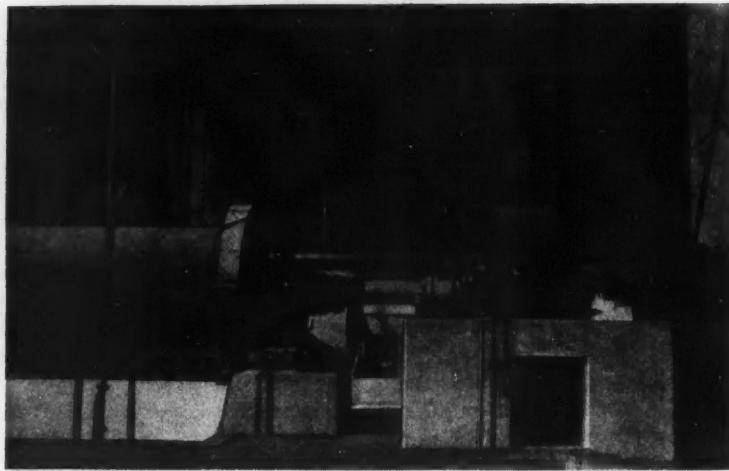


Fig. 3.

The fuel oil for firing the kiln is preheated to approximately 120 deg. C. and is atomised in the kiln through a specially designed nozzle employing air at 15 lb. per sq. in. excess pressure. As a protection against the radiant heat of the hot clinker, the pipes supplying the oil and the air blast are inside a larger pipe through which air is fed under pressure to the jet of atomised oil. At the same time this results in the oil being burnt some distance from the nozzle instead of directly at the latter. The new cooling arrangements of the "Concentra" kiln involve no shifting of the sintering zone farther up the kiln, and the flame can therefore be effectually observed from the control platform.

The kiln is pitched at an inclination of 0.36 in. to the foot. It is driven by a 35 h.p. electric motor through enclosed gearing and makes one turn in forty seconds. An overthrow device at the feed end of the kiln causes accelerated evaporation of the water contained in the slurry.

Of the two new "Centra" tube mills (Fig. 3), whose drive by a coupling spindle attached centrally to the grinding drum can be seen in Fig. 3, the raw mill gives an average performance of 110 lb. per h.p. In this mill a fineness is obtained of 6 per cent. residue on the German standard sieve "Din 70" (approximately 180 by 180 English sieve), based upon dry material and a water content of the slurry of 35 per cent. The raw materials contain a large proportion of sandy constituents which are difficult to grind. The cement mill has an average output of about 88 lb. per h.p. on sharply burned rotary kiln clinker, giving a fineness of 8 per cent. residue on the 180 by 180 sieve.

The average output of the new plant is 200 to 210 tons daily.

#### New Cement Works.

A scheme is being considered for the erection of a new cement works in Jamaica.

The Ministry of Railways in China is reported to have decided to establish a large cement works in Nanking.

#### German Cement Sales.

The sales of the Deutscher Zement Bund for the month of August 1932 were 286,000 tons, compared with 343,000 tons in August 1931 and 512,000 tons in August 1930. For the eight months January to August the sales were 1,987,000 tons in 1932, 2,784,000 tons in 1931, and 4,017,000 tons in 1930.

#### Japanese Cement "Export Guild."

The above is the title of a proposed new organisation which is being formed by the principal Japanese cement manufacturers. The suggested marketing quotas of the various companies are as follows: Asano, 37 per cent.; Onoda, 26 per cent.; Ube, 12 per cent.; Yogyo, 10 per cent.; Oita, 8.5 per cent.; Hokoku, 4.4 per cent.; Nihon, 1.3 per cent.; Iwaki, 0.8 per cent.; Tosa, 0.8 per cent.; Denkwa, 0.8 per cent. The Iwaki, Tosa, and Denkwa Kaisha concerns are permitted to market 1,500 tons a year beyond their quotas. The approximate annual capacity of the members of the proposed Export Guild is 5,600,000 tons. The new organisation will not cover Manchuria.

## Rapid Determination of Hydraulic Factors of Cement and Raw Materials.

In a recent number of "Zement," F. W. Meier points out that the rapid determination of the hydraulic factors—hydraulic, silicate and iron moduli—is of great importance in modern cement works technique, and describes new analytical methods which enable these factors to be accurately determined in a much shorter time than hitherto.

### Determination of Silica.

**IN CEMENTS AND BLASTFURNACE SLAG.**—One grammie of the substance, ground in an agate mortar and dried at 105 deg. C., is mixed with 10 c.c. of distilled water in a wide-necked Erlenmeyer flask fitted with a ground-in reflux condenser. To this is added 15 c.c. of perchloric acid reagent (containing 60 per cent. perchloric acid and 7 per cent. HCl of specific gravity 1.19), and the flask (without reflux) is heated on the sand-bath until white fumes are evolved, and then for a further fifteen minutes with the reflux attached. The hot solution is diluted with 70 c.c. of 10 per cent. HCl, digested for five minutes at 90-100 deg. C., filtered hot, washed several times with hot 10 per cent. HCl and finally rapidly with hot water, ignited, and weighed. A white pure  $\text{SiO}_2$  is obtained, the entire process taking one hour.

**IN CEMENT RAW MEAL.**—From 1.5 to 2 grammes of finely-ground material are ignited to constant weight in an electric muffle at  $800 \pm 10$  deg. C., frequently stirring with a platinum wire; 1g. is weighed into the Erlenmeyer flask and treated as before.

**IRON AND ALUMINIUM.**—The filtrate from the silica (filtrate I) is made up to 500 c.c., and 250 c.c. are used to determine the Fe, Al, Ca, and Mg. The iron is oxidised by  $\text{H}_2\text{O}_2$ , phenolphthalein is added, and the liquid is heated to 60 deg. C. and made slightly ammoniacal. The  $\text{R}_2\text{O}_3$  settles and is filtered, washing with hot water containing ammonium hydrate and nitrate. The filtrate (II) is made up to 500 c.c.

The  $\text{R}_2\text{O}_3$  precipitate is dissolved in diluted HCl and made up to 250 c.c. Iron is determined in 100 c.c. by reduction and titration with permanganate. Aluminium is determined in another 100 c.c. by precipitating the  $\text{R}_2\text{O}_3$  with a known excess of 8-oxyquinoline ("oxin") acetate solution, the excess oxin being titrated with bromine solution. The volume of oxin solution corresponding to the iron is calculated and subtracted from the total oxin used; thus the aluminium is obtained by difference.

The N/20 oxin acetate solution is prepared by Berg's method: 7.25 grammes of oxin are mixed with a few cubic centimetres of glac. acetic acid in a mortar and dissolved in hot water. After neutralisation with ammonia and cooling, this is filtered and made up to 1 litre. It is standardised against N/10  $\text{MgCl}_2$ , either by adding excess oxin and back-titrating against bromine or by the filtration

method later described (1 c.c. N/10  $MgCl_2$  = 2 c.c. N/20 oxin: the molecular weight of oxin is 145; N/20 oxin solution = 7.25g. per litre).

For the N/20 bromine solution, 4g. (1.5 to 2 c.c.) bromine and 60g. KB: are made up to 1 litre. It is standardised against the oxin solution, using as indicator a solution of 4g. indigocarmine and 2g. trinitroresorcinol per litre; excess bromine changes the colour from blue through green to yellow. One cubic centimetre of N/20 oxin = 24 c.c. N/20 bromine. For the bromine standardisation, 10 c.c. oxin are measured into a flask from a burette and 5 c.c. concentrated HCl are added to remove the faint yellow colour. Bromine is then run in until its yellow colour persists, and a drop or two of oxin is then added until the solution is again colourless. The indicator is then added (green) and bromine added drop by drop until the solution is finally yellow.

The Al determination is as follows: To 100 c.c. of  $R_2O_3$  solution (neutral or faintly HCl) are added 4g. of sodium acetate. A measured excess oxin solution is added drop by drop in the cold with shaking. On heating to 60 to 70 deg. C. the precipitate settles and is filtered, washing with cold water. The filtrate (III) is made up to 250 c.c. The excess oxin is determined in two 100 c.c. portions of III with bromide, first adding 10 c.c. concentrated HCl. For the first titration the indicator is added before titrating; the result is approximate. In the second titration the volume of bromine so found is first added, then the indicator. A few additional drops of bromine give the change from green to yellow.

The basis of the calculation is that one molecule of oxin precipitates one chemical equivalent (at. weight/valency) of  $Fe^{''}$ , Al or Mg. The method is accurate and takes only half an hour.

**CALCIUM AND MAGNESIUM.**—For these the Bucherer-Meier filtration method is used. The calcium solution is titrated under the correct conditions by N/10 sodium oxalate until no further precipitation can be observed. From 1 to 2 c.c. of reaction liquid is then removed in a pipette, filtered, and divided into two portions, one of which is tested with a few drops of N/10 sodium oxalate and the other with N/10  $CaCl_2$ , and examined for turbidity in a nepheloscope after standing for one minute. If the reaction is not complete a little more oxalate is run into the main liquid (the amount being judged from the turbidity of the test sample), and the test is then repeated. The reaction is complete when the test liquid does not show turbidity with either oxalate or  $CaCl_2$ . The precipitation is repeated on a new portion of calcium solution, using the result already obtained as a first approximation.

For calcium, 100 c.c. of filtrate II are heated to 80 to 90 deg. C. and 3 to 5 c.c. of alcohol are added. N/10 sodium oxalate is added from a burette with continuous shaking, determining the end point as above. The two titrations can be completed in 20 minutes. 1 c.c. of N/10 oxalate = 0.0028g.  $CaO$ .

Magnesium is determined in the liquid from the calcium titration by precipitation with oxin acetate solution; the calcium oxalate precipitate does not interfere. Twenty c.c. of 2N ammonium chloride and 10 c.c. of concentrated

ammonia are added to the liquid from the calcium titration, which is heated to 80 deg. C. and precipitated with standard oxin solution, determining the end point by the filtration method. 1 c.c. N/20 oxin solution = 0.001g. MgO.

**SULPHURIC ANHYDRIDE.**—This can also be determined by the filtration method. Heat 100 c.c. of filtrate I to 70 to 80 deg. C. and precipitate with N/10 BaCl<sub>2</sub> solution. The BaCl<sub>2</sub> solution is standardised by N/10 H<sub>2</sub>SO<sub>4</sub> by the filtration method. 1 c.c. N/10 BaCl<sub>2</sub> = 0.004g. SO<sub>3</sub>.

Using these methods the three important moduli can be determined in about two hours with an accuracy of about 0.1 per cent.

#### Cement Production in Spain.

Cement production in Spain during January to June 1932 was 7 per cent. less than the same period in 1931, and 23 per cent. less for this period in 1930.

#### Cement Production in Canada.

The total production of Portland cement in Canada for the period January to June 1932 amounted to 2,307,647 barrels, against 4,538,444 barrels for this period in 1931.

#### Cement Sales in Japan.

The total sales of Portland cement from Japanese-owned cement works during January to September 1932 amounted to 2,648,927 tons against 2,585,346 tons for the same period in 1931.

#### Cement Sales in Germany.

Portland cement sales in Germany totalled 720,000 tons during January to April 1932, against 1,070,000 tons for the same period in 1931. Cement exports for 1931 were 580,000 tons during 1931 compared with 950,000 tons in 1930.

#### An Australian Cement Company's Report.

The Queensland Lime & Cement Co., Ltd., showed a profit of £15,831 for the year ended July 31st, 1932, and declared a dividend of 6 per cent.; these figures compare with profits of £18,843 and £30,064, and dividends of 6 per cent. and 8 per cent. for the years 1931 and 1930, respectively.

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## Testing Paper Bags for Packing Cement.

THE strength of paper cement containers is discussed in a recent paper by Dr. G. Haegermann. The quality of paper sacks is, he states, often judged solely by tests on the paper. Although such tests are important, it is possible to have sacks of poor quality made of good paper. The quality of a sack depends on (1) the quality of the paper, (2) the number of layers of paper, and (3) the manner of construction—type of filling mouth, nature of seams and folds, and efficiency of pasting. The tests chosen must provide a criterion of the total effect of these factors.

The author studied crushing and dropping tests on sacks filled with cement. The crushing tests were carried out on sacks placed upright or horizontal and compressed between two boards in a press. The nature of the failure depends upon the position of the sack. Sacks with cross-folded bottoms, tested upright, split along the length, generally along the longitudinal fold or pasted join. Only exceptionally did the paper break in places away from the constructional folds, etc. With horizontal sacks the bottom was first forced out, followed by failure at the mouth. Only under considerably higher loads did the sacks fail along the longitudinal fold or where the length of the sack came into contact with the horizontal support. The sacks have already failed, of course, when the bottom is first ruptured. For sacks tested in an upright position failure occurs at about 1,600 lb. pressure. For sacks tested horizontally, in one manufacture the bottom failed at about 2,000 lb., the mouth at 6,000 lb., and the sides when the pressure was increased to 25,000 lb. For another brand of sack the corresponding average figures were 3,500 lb., 6,500 lb., and 22,000 lb.

The results of crushing tests are not, however, of direct practical value, since when sacks are stored the bursting of the base is largely prevented by the pressure of neighbouring sacks. Dropping tests are of greater value.

In the dropping tests filled sacks were dropped from determined heights, from upright and horizontal positions, on to a concrete floor. In the upright tests the mouth of the sack, pointing upwards, was held in a clamp which was released to drop the sack. In the horizontal tests the sack rested on raised trap-doors which fell apart at the middle to release the sack. The types of failure in these tests were similar to those in the crushing tests. In the horizontal tests the bottom or mouth was ruptured. In the upright tests the sacks generally burst along the longitudinal fold, sometimes also along the pasted join, and only rarely in other places.

The method of test finally adopted was to use a constant drop of 30 in. for the horizontal tests and 20 in. for the upright tests, repeating the drop until failure occurred. The results often vary considerably and the mean value for ten sacks (or at least five) should be taken. The results of crushing and dropping tests and of the usual paper tests for three types of sack show that the ordinary tests on paper do not alone afford a criterion of the quality of the sacks. Wired sacks appear to be superior to sacks with cross-folded bottom. Folding the paper has a considerable effect on the resistance of the

sacks. Folds are, of course, necessary, but sharp folds should be avoided. Pasting the separate layers of paper together at isolated points is also disadvantageous.

Dropping tests with sacks filled with the prescribed quantity of cement are best, using 30in. fall for horizontal and 20in. for vertical sacks. Good sacks should not be ruptured by the first fall. The number of falls to rupture affords a criterion of the quality of sacks of different makes.

With a 30in. fall of horizontal sacks, from four to six drops were sustained before failure occurred, and with a 20in. fall of upright sacks bursting took place with from two to six drops.

Four-ply paper bags wired at both ends withstand twelve horizontal falls of 30in. or fourteen vertical falls of 20in. before rupture, while three-ply bags with cross-folded bases and wired mouths withstand from one to four drops similarly applied.

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#### Cement Exports and Imports in China.

Imports of Portland cement into China during January to August 1932 totalled 156,002 tons, against 195,764 tons for the same period in 1931. Exports were 19,329 tons during January to August 1932, against 26,426 tons for the same period in 1931.

### NOTICE.

#### **PAPER SACKS.**

*In an action in the Chancery Division brought against B. Kershaw & Co. (1920) Limited to restrain infringement of Bates' Patents Nod. 252038x, 250917 and 251016 protecting "open-mouthed" and "valved" sewn paper sacks and machinery for making same, the Defendants on the 9th December, 1932, submitted to an injunction in respect of all three patents.*

*The above patents are owned by Paper Sacks Limited of Northfleet, Kent, and it is their intention to enforce their rights against all persons attempting to infringe.*

**PAPER SACKS LIMITED.**

## Recent Patents relating to Cement.

### Cement Compositions

379,320.—Lagas, A. J., 49, Duvelandschestraat, The Hague. Feb. 27, 1931.

A cold cement glaze composition consists of cement to which is added soap solution, a solution of alkaline earth metal salt such as magnesium silicofluoride or calcium chloride, and a small proportion of an organic oxyacid such as tartaric or citric, or polybasic acid such as oxalic, or a salt thereof, with, if desired, slaked lime. Linseed oil soap is preferably used. In an example 400 gm. of linseed oil soap are mixed with 4 litres of water and 75 gm. of slaked lime in 1 litre of water. The mixture is added to 5 litres of 30 per cent. magnesium silicofluoride and rubbed through a sieve with 900 meshes per square centimetre. After dilution in 30 litres of water, 1 gm. of cream of tartar, or potassium oxalate, or sodium citrate is added and the mixture added to Portland cement.

### Cements and Mortars

371,770.—Chemische Fabrik Grünau, Landschoff & Meyer Akt.-Ges., Grünau, Berlin. Nov. 6, 1931.

Thiocyanic acid or one or more salts thereof is added to cement, mortar, or concrete to regulate the setting or hardening and to increase the density thereof. Ammonium, calcium, potassium, barium, aluminium, iron, and chromium thiocyanates are specified; they may be used alone or in conjunction with other salts such as chlorides, nitrates, etc. The additions may be made either to the dry cement or when mixing the concrete or mortar. According to examples, a Portland cement mixed with a 21 deg. Bé solution of calcium thiocyanate sets in 10 to 20 minutes. If equal proportions of calcium and aluminium thiocyanates are used, setting takes place within 5 minutes and the temperature rises by 40 deg. C.

### Plaster Compositions

374,455.—Lefebure, V., Willett Building, Sloane Square, London, and Imperial Chemical Industries, Ltd., Millbank, London. Feb. 4, 1931.

A plaster-cement mix which sets with a glossy surface consists of 5 to 95 per cent. hydrated lime, or Portland, white or aluminous cement with 95 to 5 per cent. plaster of the accelerated anhydrite type, with or without fillers such as sand or pigments. If applied to absorbent backings, the backing is preferably first rendered non-absorbent, e.g., by spraying with water or applying an aqueous bitumen emulsion. Hygroscopic agents such as calcium chloride or glycerol may also be added to the wet mix. When

spraying, the mixture is preferably ground to pass through a 200-mesh sieve or may contain about 50 per cent. of a relatively coarse filler such as sand. In an example 90 parts of ground anhydrite (catalysed with 1 per cent. of potassium sulphate) and 10 parts of white cement and if desired, pigment, are gauged with 35 per cent. of water and sprayed on a cement-sand coating previously applied to a brick wall, which may be previously treated with a thin film of aqueous bitumen emulsion. In another example a mix is poured or sprayed into porous tile moulds the base of which is kept moist by standing them in  $\frac{1}{2}$  in. of water. The moulds in this case may be made of the same material as the mixture which is sprayed into them.

### Cement Manufacture

378,555.—Krupp Grusonwerk Akt.-Ges., Buckau, Magdeburg, Germany. May 20, 1931.



In the manufacture of cement, the raw slurry, before delivery to a rotary kiln (5) is dehydrated in a filter (1) and then passed in succession through a drier (2) and a rotary preheater (4), both heated by the waste gases from the kiln (5) and so arranged that the whole of the waste gases pass first through the preheater and then through the drier. The filter (1) is of the kind comprising a drum around which pass cords or wire fabric bands (3), the latter extending into the drier (2) which is fitted with a shaking or knocking device to remove the filtered material from the bands.

### Recent Patent Applications

373,796.—B. MOORE: Refractory cements.

374,455.—V. LEPEBURE and IMPERIAL CHEMICAL INDUSTRIES, LTD.: Plaster and cement mixes and methods of application.

373,248.—C. PONTOFFIDAN: Manufacture of rotary kilns for cement.

375,045.—A. WEITHALER: Iridescent effects on cement or like coatings and surfaces.

375,144.—H. PLANSON: Manufacture of artificially coloured cement, plaster, etc.

381,133. F. L. Smith & Co. Manufacture of cement.

### Book Review.

**Zement Kalender, 1933.** Charlottenburg: Zementverlag, G.M.B.H. Pp. 430.  
Price, RM. 3.20.

THIS book contains a good more than its title implies, because in addition to the usual tables of weights and measures, logarithms, densities of materials, etc., that are to be found in most pocket-books there is sufficient other matter to warrant it being described as a text-book on concrete.

The German Standard Specification for Portland cement is given in full, with the modifications that apply to the slag cements that are more prominent in Germany than elsewhere. The various binding materials, ranging from rapid-hardening Portland cement down to gypsum plaster and lime, are defined, the methods of manufacture outlined, and the distinctive qualities described in detail. Attention is given to the combination of materials for plastering and the effects of extremes of temperature and chemicals upon concrete. A chapter is devoted to the general principles of concrete and reinforced concrete, some information on labour costs being included. Other sections deal with pre-cast concrete, partitions, asbestos cement, and concrete roads.

The regulations of the German Commission for Reinforced Concrete (May 1932) are given in detail and constitute a small treatise on the subject. Together with formulæ for reinforced concrete calculations and information on such subjects as concrete in sea-water and peaty water, slag aggregate, and light-weight aggregate, the book should be useful to those concerned with concrete and to whom the language is no obstacle.

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